

DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

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UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR
SODIUM CARBONATE, GRANULAR (MONOHYDRATE CRYSTALS)

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 432

This specification was officially promulgated by the Federal Specifications Board on September 25, 1926, for the use of the departments and independent establishments of the Government in the purchase of sodium carbonate, granular (monohydrate crystals).

[The latest date on which the technical requirements of this specification shall become mandatory for all departments and independent establishments of the Government is December 27, 1926. They may be put into effect, however, at any earlier date after promulgation]

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I. GENERAL SPECIFICATIONS

There are no general specifications applicable to this specification.

II. GRADE

The material shall be of one grade only, as hereinafter described.

III. MATERIAL

See General requirements.

IV. GENERAL REQUIREMENTS

Sodium carbonate, granular, shall be a granular, high-grade sodium carbonate with 1 molecule of water of crystallization ($\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$).

V. DETAIL REQUIREMENTS

Sodium carbonate, granular, as received shall conform to the following requirements:

1. Total alkalinity, calculated as Na_2O , shall be not less than 48.5 per cent, equivalent to 83 per cent of anhydrous sodium carbonate (Na_2CO_3).
2. Hydroxide (NaOH) shall not be present.
3. Bicarbonate shall not be present.
4. Matter insoluble in water shall not exceed 0.1 per cent.
5. It shall be granular.

VI. METHODS OF INSPECTION, TESTING, AND BASIS OF PURCHASE

1. SAMPLING

(a) WHEN PACKED IN CANS OR CARTONS.—One can or carton shall be taken at random from not less than 1 per cent of the vendors' shipping containers, provided such containers contain not less than 50 pounds each. In the case of smaller containers a can or carton shall be taken at random from each lot of containers totaling not to exceed 5,000 pounds. The total sample shall in all cases consist of not less than three cans or cartons taken at random from separate containers. With very large lots, where the sample drawn as above will amount to more than 20 pounds, the percentage of packages sampled shall be reduced, so that the amount drawn shall not exceed 20 pounds. Wrap the individual cans or cartons tightly in paraffined paper at once and seal by rubbing the edges with a heated iron. The inspector should accurately weigh each wrapped can or carton, record its weight and the date of weighing on the wrapper, place the wrapped cans or cartons in an air-tight container, which should be nearly filled, seal, mark, and send to the laboratory for test. Samples should be kept cool until tested. The seller shall have the option of being represented at the time of sampling, and when he so requests shall be furnished with a duplicate sample.

(b) WHEN IN BULK.—A grab sample of not less than one-half pound shall be taken at random from not less than 1 per cent of the vendors' shipping containers, provided such containers contain

not less than 100 pounds each. In case of smaller containers a grab sample of not less than one-half pound shall be taken at random from each lot of containers totaling not to exceed 10,000 pounds. The total sample shall in all cases consist of not less than three grab portions taken at random from separate containers. With very large lots, where the sample drawn as above will amount to more than 20 pounds, the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 20 pounds. The inspector should rapidly mix the sample, place in an air-tight container, which shall be filled; seal, mark, accurately weigh, record its weight and date of weighing on the package, and send to the laboratory for test. Samples should be kept cool until tested. The seller shall have the option of being represented at the time of sampling and, when he so requests, shall be furnished with a duplicate sample.

2. TESTING

(a) **PREPARATION OF SAMPLE.**—Rapidly mix the sample, if desired, quarter down to about 1 pound, and weigh out all portions for analysis at once. Unused portions of the sample used for analysis shall be preserved in an air-tight container in a cool place. Note the condition of the sample.

When a determination shows nonconformity with specification, a duplicate shall be run.

(b) **PRELIMINARY PROCEDURE.**—Transfer 5 g of the sample to a 1-liter volumetric flask, add about 200 cc of freshly boiled and cooled distilled water, and when the sample has dissolved, dilute to the mark with freshly boiled and cooled distilled water, and mix. To about 10 cc of this solution in a beaker add a 10 per cent solution of barium chloride until a precipitate no longer forms, filter off the precipitate, and add a drop of 10 per cent silver nitrate solution to the filtrate. A dark coloration indicates the presence of a hydroxide.

(c) **TOTAL ALKALINITY.**—To a 200 cc aliquot of the above solution (corresponding to 1 g of the sample) add three drops of methyl orange solution and titrate quickly to the end point with 0.5 *N* H_2SO_4 . Then take another 200 cc aliquot of the solution and quickly add about 1 cc less of the standard acid than was required in the previous titration, avoiding loss by effervescence. Cover the beaker with a watch glass and boil the solution to expel free CO_2 . Cool, wash off cover glass, add three drops of methyl orange solution and complete the titration, adding the standard acid dropwise and stirring thoroughly. Calculate the percentage of total alkalinity as Na_2O (1 cc 0.5 *N* H_2SO_4 = 0.0155 g Na_2O).

(*d*) HYDROXIDE.—If the sample contains hydroxide, add to a 200 cc aliquot of the original solution 100 cc of a 10 per cent solution of BaCl_2 and stir thoroughly. Filter off the precipitate and titrate the filtrate at once with 0.5 *N* H_2SO_4 , using phenolphthalein as indicator. Calculate the percentage of hydroxide as NaOH (1 cc of 0.5 *N* acid=0.02 g NaOH). If the sample contains hydroxide it will not contain bicarbonate.

(*e*) BICARBONATE.—Titrate a 200 cc aliquot of the original solution with 0.5 *N* NaOH until a drop of the solution added to a drop of 10 per cent AgNO_3 solution on a spot plate produces instantly a dark coloration. Calculate the percentage of sodium bicarbonate (1 cc of 0.5 *N* NaOH=0.042 g NaHCO_3).

(*f*) CARBONATE.—Subtract the number of cubic centimeters of 0.5 *N* reagent required for the hydroxide or bicarbonate from the number of cubic centimeters of 0.5 *N* H_2SO_4 required for the total alkalinity and calculate the difference to percentage of Na_2CO_3 (1 cc of 0.5 *N* solution 0.0265 g Na_2CO_3).

(*g*) MATTER INSOLUBLE IN WATER.—Transfer 10 g of the sample to a 600 cc beaker, add about 400 cc of freshly boiled distilled water, and boil the solution for 10 minutes. Filter on a weighed Gooch crucible, wash thoroughly with hot water, dry the crucible and residue at 105 to 110° C. for three hours, cool and weigh. Calculate the percentage of matter insoluble in water.

3. REAGENTS

(*a*) STANDARD SODIUM HYDROXIDE SOLUTION.—0.5 *N*, or about 20 g, of pure sodium hydroxide dissolved in water and diluted to 1 liter. Standardize against Bureau of Standards standard benzoic acid, using phenolphthalein indicator.

(*b*) STANDARD SULPHURIC ACID SOLUTION.—0.5 *N*, or about 25.8 g, strong sulphuric acid (specific gravity 1.84) diluted to 1 liter. Standardize against standard sodium hydroxide solution (*a*), using methyl orange indicator.

(*c*) BARIUM CHLORIDE SOLUTION.—Dissolve 100 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in distilled water and dilute to 1 liter.

(*d*) SILVER NITRATE SOLUTION.—Dissolve 10 g of silver nitrate in water and dilute to 100 cc.

(*e*) METHYL ORANGE INDICATOR.—Dissolve 1 g of methyl orange in 1 liter of distilled water.

(*f*) PHENOLPHTHALEIN INDICATOR.—Dissolve 1 g of pure phenolphthalein in 100 cc of 85 to 95 per cent ethyl alcohol.

4. BASIS OF PURCHASE

Sodium carbonate, granular, shall be purchased by net weight.

VII. PACKING

Packing shall be in accordance with commercial practice, unless otherwise specified.

VIII. NOTES

Sodium carbonate conforming to this specification is suitable for photographic purposes.

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